A STUDY OF MATERIALS AND TECHNIQUES FOR THE CONSERVATION OF TWO MINIATURE PAINTINGS

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Keywords: oil painting, deterioration, safflower oil, alla-prima, conservation,

1. Introduction

The conservation of oil paintings is an accurate technical process requiring an extensive knowledge of many sciences and an understanding of different disciplines and applied sciences in order to ensure the correct restoration and conservation of damaged oil paintings and so preserve these works of art in the best possible manner.

The investigation of an artwork to understand its history, state of conservation and to gain knowledge about the constituent materials, is an issue that is obtaining growing attention in specialized literature. Therefore, assessment of the preservation status must be conducted in an objective way by analyzing the integrity of the materials and examining and describing any changes in the physical-chemical properties, quantitatively.

This involves photography using different light sources: visible, ultraviolet (UV) and infrared (IR), in order to highlight iconographic details, conservation problems, painting techniques and preparatory drawings. SEM-EDAX and XRD are used to identify the pictorial palette pigments and inorganic additives in the preparation layer. In this study, micro-samples of materials were taken and analyzed by Infrared Spectroscopy (FT-IR) and a UV Spectro Photometer, to identify the organic pigments and the sizing coat (isolation layer); some samples were also analyzed by GLC to identify the oil medium.

Information about the materials constituting an artwork and their distribution, together with knowledge of the phenomena occurring at the interface with the environment, can lead to proper interpretation of any alterations that may have occurred due to natural or accidental events. This can also lead to a better understanding of the executive techniques and conservative history of the object through the identification and recognition of any previous interventions and of the preservation status of the artwork. In addition, the identification of newly formed products can provide input for an appropriate restoration project [1].

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There are certain forms of deterioration which must be treated prior to any intervention regarding the detachment procedure, while other forms need to be handled after the detachment procedure.

The two oil paintings under examination are supported on paperboard and are both fixed to a single secondary paper support. In this case, separation of the two paintings was required. Before separating the two images, certain precautions had to be taken and, imperatively, be applied. This was necessary so as to maintain the integrity of the paint layer during and after the process of separation, as well as during the steps in reconstructing the different layers.

In carrying out the restoration and conservation of oil paintings, we highlight that it is essential to fully understand all the different domains of the applied sciences before starting the practical steps needed for their restoration and conservation.

2. Historical Studies

This research includes a study of two miniature paintings, registered at the Museum of Helwan University, Faculty of Applied Arts, Giza, Egypt, No.(A) of 100/6. The two paintings are unsigned and undated.

The two oil paintings are supported on paperboard and are both fixed to a thick secondary paper support. One of them represents a noble sitting on a carpet and in front of him stands a maid. The painting is decorated with floral and geometrical ornamentation. According to the artistic and pictorial characteristics of the individuals portrayed in the picture (Figure 1a), the painting follows the Persian style and dates back to the Zand dynasty (prior to 1779). It represents the end of the Safavid era, as we can observe by its style and form; for example, the lights and shadows of the human figures are close to the Qajarian style [2]. The second painting, conceptually, resembles the former and dates back to the same period, but the style used is that of Central Asia (Figure1b).

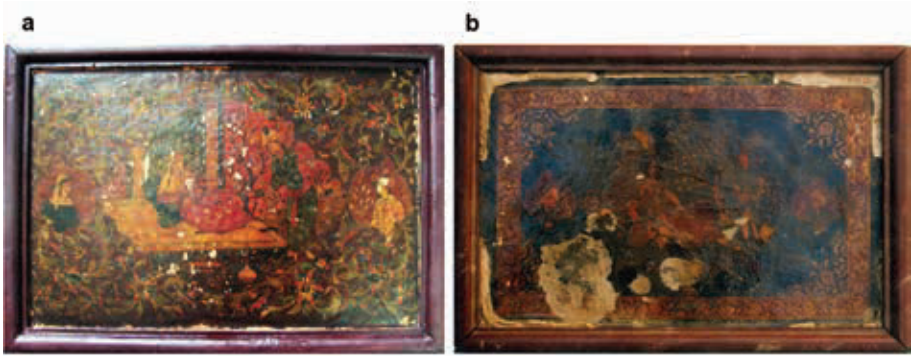

Figure 1. a) Persian Style miniature painting, No.(A) of 100/6 from the Faculty of Applied Arts Museum, Giza, Egypt, before conservation; b) Central Asia book cover painting, No.(A) of 100/6 from the Faculty of Applied Arts Museum, Giza, Egypt, before conservation.

3. Materials and Methods

3.1 Optical Light Microscope (OLM) Examination

For stratigraphic analysis, a cross-section of these paintings was examined with a Zeiss standard stereomicroscope of the stemi DRI663 type, using reflected light. Vertical photo microscopy (VPM) was used to study the direction of the brushstrokes [3], as well as invisible damage such as micro cracks and flaking in the paint layer [4]. The conservation state of the paperboard was also examined, and a Herzberg Stain test was performed to identify the pulp [5].

3.2 Investigation Using UV and IR Lamp

UV radiation was used to detect deterioration and past restoration areas, if any, of the paint layer (Figure 2a). Moreover, IR radiation was used to study the artistic characteristics of the painting and forms of deterioration by means of a different method (Figure 2b).

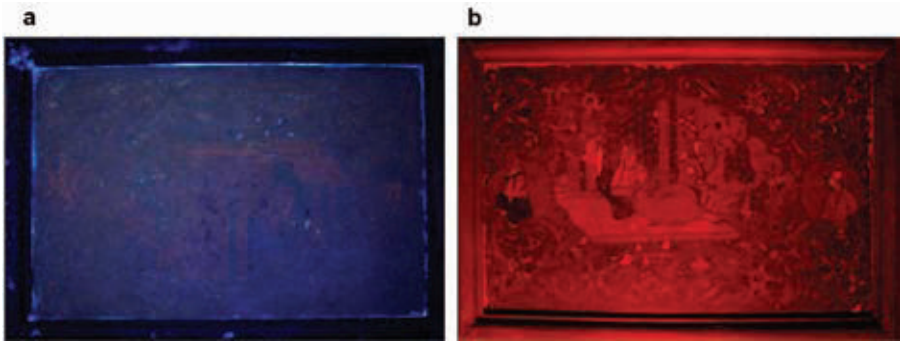

Figure 2. a) UV Investigation; b) IR Investigation.

3.3 Analysis Using XRD

X-Ray diffraction (XRD) was used to analyze the inorganic materials in the paint layer and to identify the fillers and additives in the paperboard. Philips X-Ray diffraction equipment, model PW/1710 with Co radiation, Fe filter at 40 KV, 30 MA and scanning speed 0.02/s was used. From the diffraction charts, the dA and the relative intensities were obtained and compared with JCPDS (Joint Committee on Powder Diffraction Standards) files [6].

3.4 Investigation and Analysis Using SEM – EDAX

Investigation and analysis with a scanning electron microscope equipped with an Energy Dispersive X-Ray analysis (EDX) unit was carried out using a Philips XL30 KV model and resolution for W (3.5nm), to identify the constituent elements of the inorganic pigments and their proportions, as well as the form of the granules of the pigment crystals [7].

3.5 Analysis Using UV Spectro Photometer

Analysis with a UV Spectro Photometer was used to analyze the organic materials in the paint layer and varnish. A UV Spectro Photometer λ Lambda 900 Peckin Elmer Double Beam was used.

3.6 Analysis Using Fourier Transform Infrared Spectrometer / FTIR

FTIR was used to identify the sizing coat (isolation layer) and the organic binding medium in the paint layer. A JASCO FT / IR 6100 Spectrometer was used.

3.7 Analysis Using Gas Liquid Chromatography (GLC)

GLC was used to identify the oil medium in the paint layer. GLC is considered to be one of the most successful techniques for determining the fatty acids in the binding medium and studying the changes which take place during the aging process [8].

4. Results and Discussion

4.1 Painting Technique

4.1.1 Results of the cross-section examination of samples from the first painting as shown in Figure 3a

The cross-section examination of the brown background (S1) under the optical light microscope (125X) revealed two paint layers; the lower one was thick and light colored, while the upper one was very fine and brown. It did not reveal any ground layer (Figure 3c). Specifically, the cross-section of the red area (S2) of the painting revealed two layers: the upper one was semi-thick and red, while the lower layer was thick and green (Figure 3d). The cross-section of the green area (S3) of the painting, showed one layer of green color (Figure 3e). The number of layers confirms that the artist used the *alla-prima* technique and that the paint layer was applied directly to the paperboard.

4.1.2 Results of the cross-section examination of samples from the second painting as shown in Figure 3b

The cross-section of the black background (S4) under the optical light microscope (125X) revealed that the first paint layer was very fine and black (Figure 3f). Further examination did not reveal any ground layer. The cross-section of the red area (S5) of the painting revealed one semi-thick red layer (Figure 3g). The cross-section of the gilded area (S6) of the painting consisted of two layers: the upper was very fine and gilded, while the lower one was semi-thick and red (Figure 3h). The number of layers confirms that the artist used the *alla-prima* technique and that the paint layer was applied directly to the paperboard.

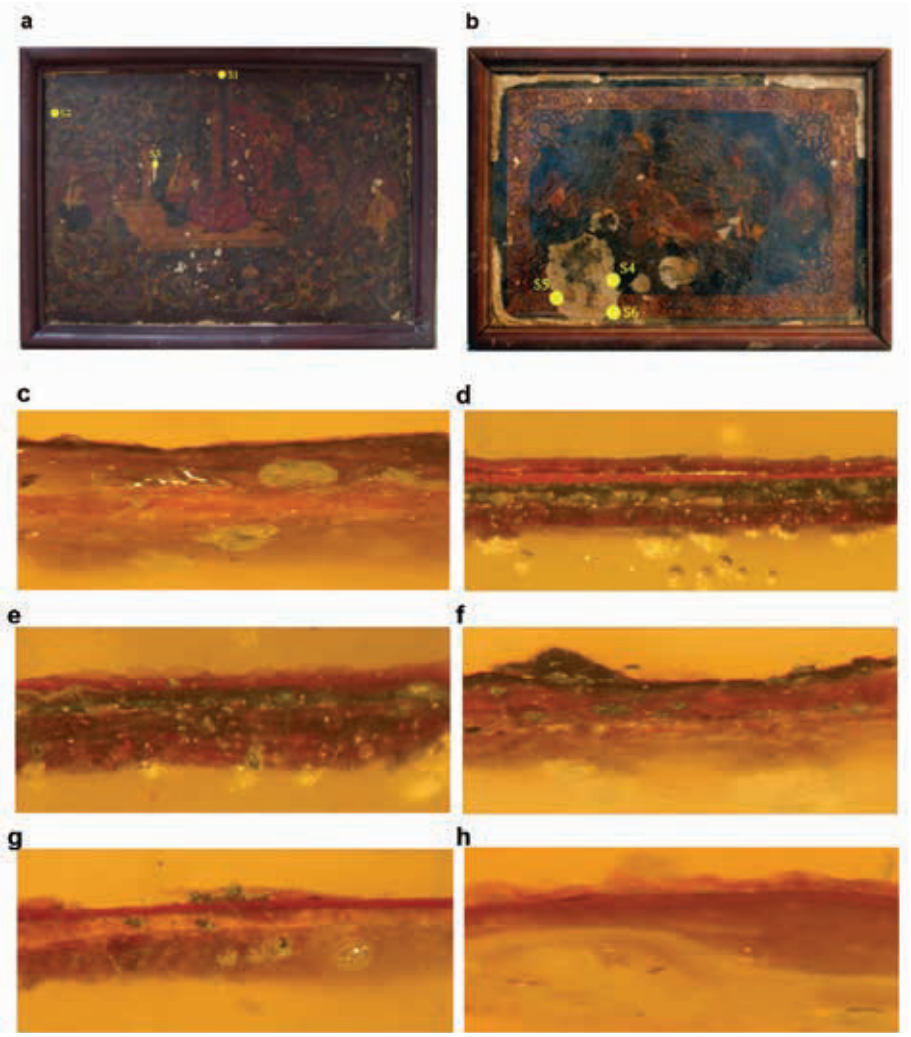

Figure 3. a) Cross-section samples of first painting: cross-section examination of the brown background S1 (c), cross-section examination of the red area S2 (d), and cross-section examination of the green area S3 (e). b) Cross-section samples of second painting: cross-section examination of the black background S4 (f), cross-section examination of the red area S5 (g), and cross-section examination of the gilded area S6 (h).

4.2 Paint Layer Details

The suggested method (VPM) was used to study the techniques and characteristics of the two paintings.This method showed that the painter used two or three types of brushes to apply the paint layer and that the brushstrokes went in all directions (Figure 4a). The use of VPM showed that the paperboard was of a smooth type (Figure 4b) and also revealed the presence of white salt crystals (Figure 4c, d) [9].

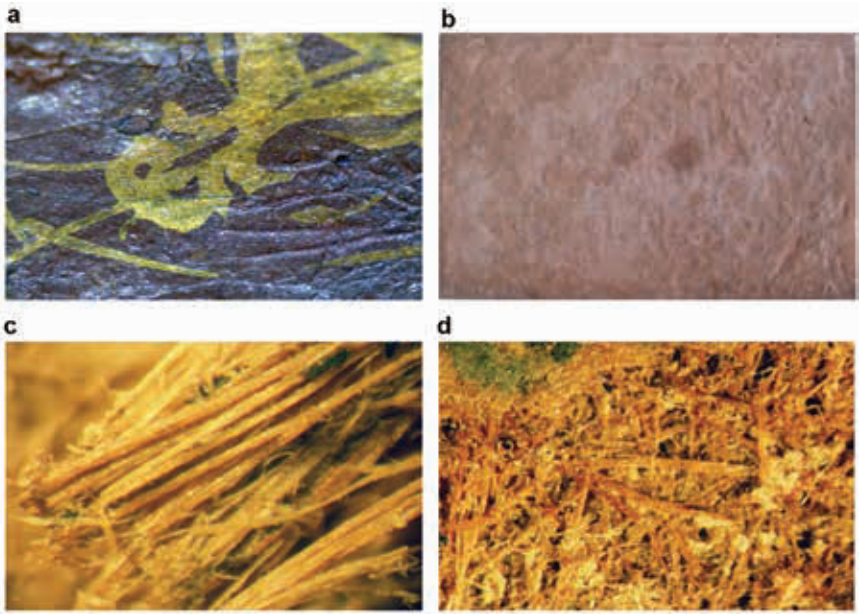

Figure 4. a) Details of the paint layer; b) paperboard of smooth type; c) microscopic image of the secondary support; d) presence of white salt crystals in secondary paper support.

4.3 Pigments Used in Paint Layers of Both Paintings

Results revealed that lead red (Pb3O4) was used as a red pigment in painting No. 1(Persian Style miniature painting) (Standard No. 8-19 JCPDS) (Figure 5a, b).

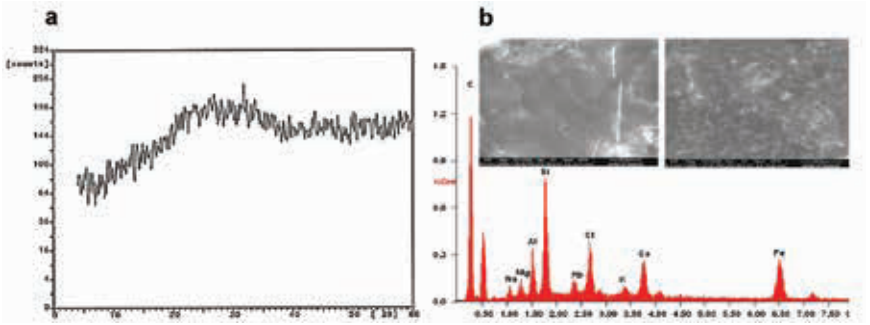

Figure 5. a) XRD result of red sample; b) SEM - EDAX result of red sample.

4.3.1 Red Pigment from Madder

UV Spectro Photometer analysis also revealed that madder was used as a red pigment in painting No. 2 (Central Asia book cover painting) (Figure 6a). Madder is one of the most important red dyes and pigments derived from the roots of the madder plant, Rubiatinctorum [10].

Madder was used in many parts of Europe, where this plant was grown, such as in Normandy, Languedoc, Spain, Sicily, Lombardy, etc. It contains red and yellow dyes [11], depending on the basic components.The dye is composed of two types of components: the first, alizarin, purpurin, pseudo-purpurin; the second, alizarin 2-methylether, rubiadin, munjistin [12]. While the former components give a red color, the latter group of components give a yellow color [13]. There are also different types of madder plant (Rubiatinctorin L.), Lady's Bedstraw (Galliumborede, Goverum L.), Wild Madder (Rubiapergrina L.), Indian Madder (Rubiamungista L.) [13].

4.3.2 Black Pigment from Tannin

UV Spectro Photometer analysis revealed that tannin was used as a black pigment in painting No. 2 (Central Asia book cover painting) (Figure 6b). Tannin is obtained from the fruits, roots, bark and wood of a number of plants and trees.Well-known sources of tannin include oak galls, Sumach leaves and divi-divi fruits.The basic components of tannin are gallic and di- and tri-gallic acids, including ellagic acids. Tannin is a very acidic organic substance due to the presence of phenolic, hydroxyl and carboxyl groups in their chemical structure [13].

Tannin is divided into gallotannins and ellagitannins, and can be brown or black when iron is added to it. Moreover, tannin is used for ferro-gallate inks [14].

4.3.3 Brown Pigment from Logwood

UV Spectro Photometer analysis revealed that logwood was used as a brown pigment in painting No.1(Persian Style miniature painting) (Figure 6c).

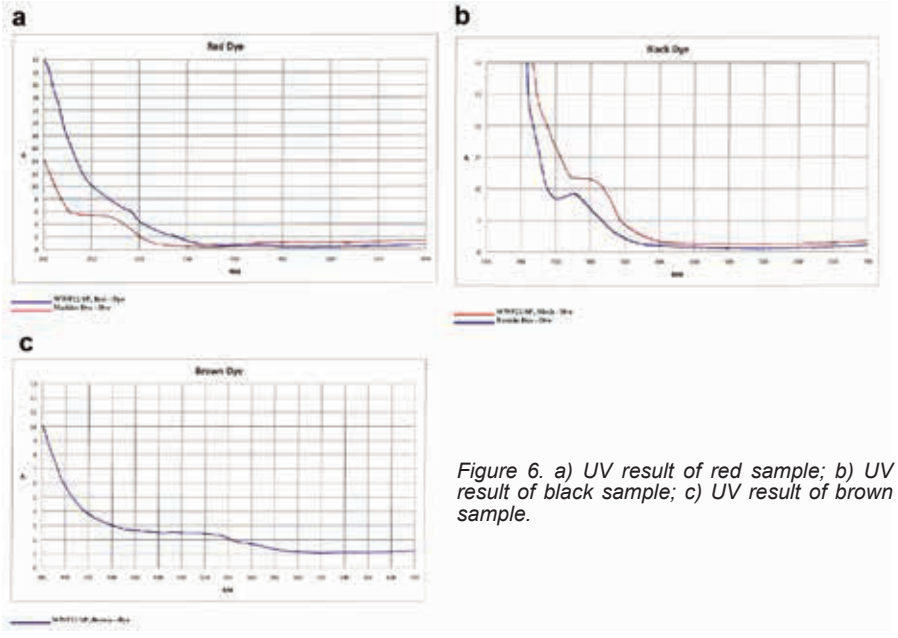

Figure 6. a) UV result of red sample; b) UV result of black sample; c) UV result of brown sample.

The logwood gives either a dark reddish orange colour or sometimes, a dark violet or purple colour, and can also turn into light yellow over time until it becomes black. Logwood is also called Campeachy Wood, Blackwood, Bois Blue, Blauholz. It grows in Central, North and South America, and reaches up to 30-45 feet in height. The tree bark is smooth and flat, and is dark brown in colour; the flowers are yellow and its seeds look like beads in the shape of a double kidney [15].The artist in this case applied the method of “half-tones», a method that is dependent on one important characteristic: the color and its intensity or strength of coverage, where the artist applied a red layer over a black layer to produce a brown layer.

4.4 Medium Used in Paint Layers

The GLC analysis of samples taken from the paint layer of these paintings showed the presence of a small amount of saturated fatty acids and detectable levels of palmitic and stearic acids. The interpretation of these results indicates the presence of safflower oil (Figure 7).The results of the P/S ratio of palmitic to stearic acids determined that safflower oil was used as the oil medium in the paint layers.

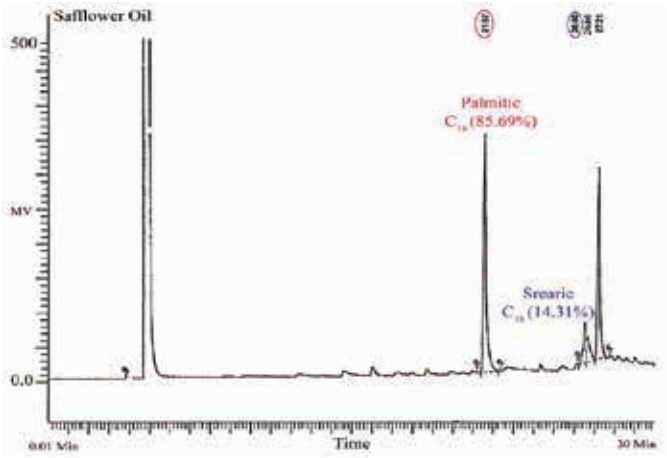

Figure 7. GLC result of oil medium.

4.5 Sizing (Isolation Layer)

FTIR analysis is a method used to identify the glue (sizing) on the paperboard. The interpretation of the FTIR spectrum revealed that animal glue was used as the sizing coat (Figure 8).

A sizing coat is considered either as a ground layer in these paintings or as an isolating layer. The interpretation of the presence of a proportion of oil with animal glue in the sizing coat does not mean that the artist mixed the oil with the solution of colloidal to make the isolation layer, it is due to the fact that a percentage of the oil had moved from the paint layer to the sizing coat. The main goal of the sizing was, moreover, to isolate and protect the paper support from the paint layer, whether oils or pigments were used [16].

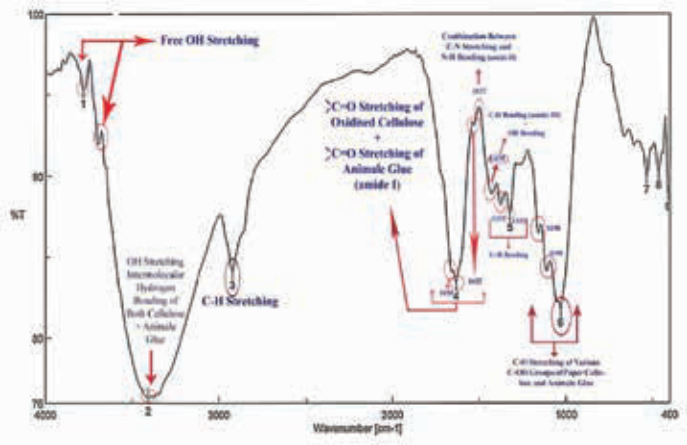

Figure 8. FTIR result of sizing coat and paperboard.

4.6 Varnish Analysis

UV analysis was used to identify the type of varnish. The analysis showed that the varnish used in these paintings was Arabic gum (Figure 9).

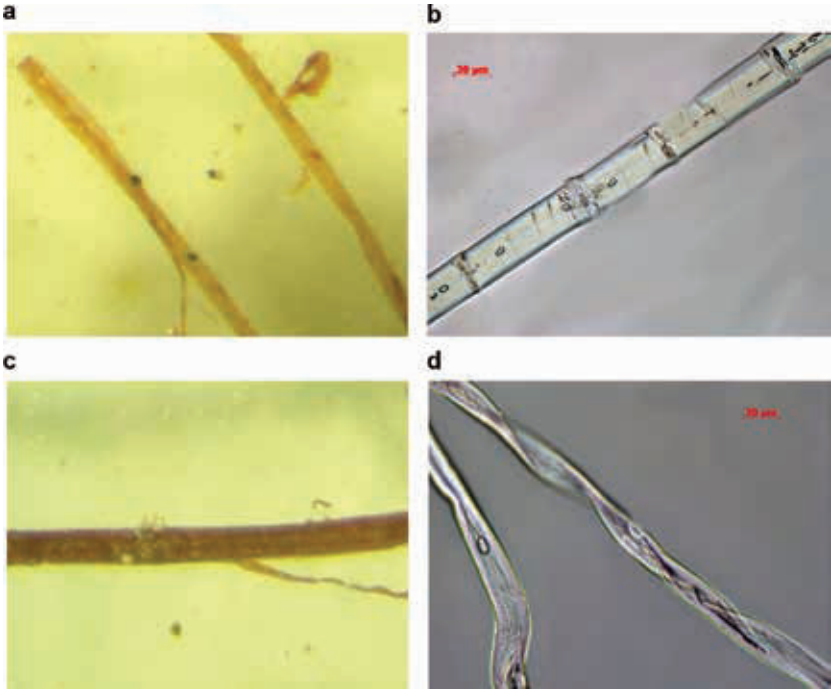

Figure 9. UV result of varnish type.

4.7 Paperboard Support

The result of examination using Optical Light Microscopy (OLM) showed that the pulp used in the paperboard is rag paper made from cotton and linen fibers (Figure 10).

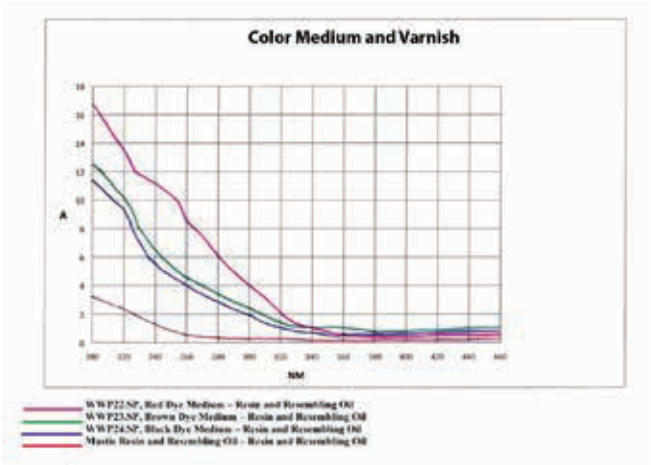

Figure 10. a) Microscopic image of the cotton fiber from the paperboard pulp; b) SEM examination of the cotton fiber from the paperboard pulp; c) a microscopic image of the flax fiber from the paperboard pulp; d) SEM examination of the flax fiber from the paperboard pulp.

XRD analysis results also showed that fillers or additives were used between the fiber pulp rags. They were calcite (calcium carbonate) CaCO3, (Standard No. 05-586 JCPDS), aluminum silicate AlSiO2 (Standard No. 37-1460 JCPDS), quartz SiO2 (Standard No. 46-1045 JCPDS), topazlite CaO.Fe2O3.3SiO2 (Standard No. 3-1135 JCPDS) (Figure11).

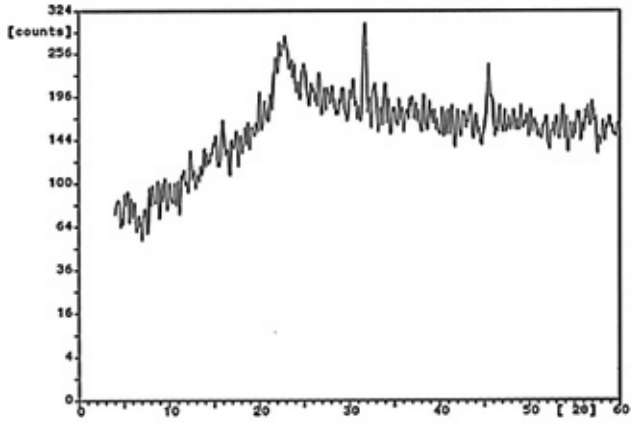

Figure 11. XRD result of the paperboard analysis.

4.8 Conservation State of Paintings

These paintings have been exposed to severe conditions, improper storage and neglect which together have caused several deterioration phenomena, i.e. tears, darkening of the varnish layer, cracks, dirty appearance, scratches, lacunas and brittleness of the paperboard. The intensity of the colours has been lost due to the dirty appearance and the darkened varnish; however, the original intensity of the colours is preserved under the frame. Two lacunas were found. The abrasion, tears, cleavage and flaking can be easily noticed. The support is brittle and discoloured, it has turned into a greyish brown; and the PH value is 5.5 (Figure 12). PH is the prime indicator of paper permanence, not lignin [17]. When the oil medium is exposed to environmental action or the passage of time, it deteriorates, and the fatty acid ratio also changes [18]. Weak adhesion between the components of the painting and partial dissolution of the sizing material between the paint layer and the support, as a result of direct contact with water, have also caused several deterioration phenomena. The darkening of the varnish is mainly due to the long-term exposure of the painting to ultraviolet radiation and the presence of particulate matter in the atmosphere.

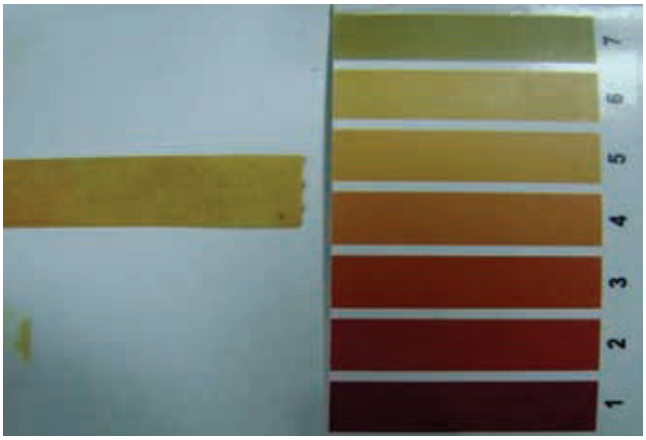

Figure 12. Showing PH value of the paperboard.

5. Restoration and Conservation Processes

There were forms of deterioration which had to be treated prior to any intervention regarding the detachment procedure and other forms which needed to be handled after the detachment procedure. The steps used in the treatment, detachment and remounting of the oil painting on the new support were as follows.

First, the painting was separated from the frame, the dirty darkened varnish layer was removed using ethyl alcohol which gave successful results [19].

To consolidate and improve the adherence of the paint layer and the ground to the paperboard, a solution of 5% and 10% of Beva 371 in white spirit was applied by brush, and then square pieces of Japanese paper and gauze were applied using Beva 371,

this was done to reinforce the paint layer and to provide protection for the paint layer during the treatment procedure on the back of the painting. The detachment was carefully carried out using handsaw, scalpel and cauter without causing any stress.

The cracks, especially those located in the upper left part, were fixed by using the cauter [20].

To restore lacunas on the board, a piece of special acid free board was inserted in the lacunas and fixed to it by the "window method". The thickness was the same as the thickness of the paperboard. To de-acidify the paperboard, it was brushed with 2.5% magnesium carbonate with distilled water [21] which increased the PH value to 7.

To mend the tears in the board, the edges of the tears were brushed with 10% water solution of rabbit skin glue, the edges were then repaired using a cauter. In order to consolidate the board, 5% of Plextol B500 was applied by brush.

For the mounting, the original painting support was consolidated by spreading a layer of Plextol B500 on the verso of the painting, and then Japanese paper of the same board size was glued on the back of the painting board by using 5% Plextol B500. Some Japanese paper strips were also glued on the edges of the board; an acid free board was then used to mount the original board using Plextol B500. The painting was subsequently placed on the new support.

Toluene was used to remove the cushion layers of Japanese paper and gauze from the paint layer. The flaking paint layer was additionally pressed to the board. An emulsion fill was used on the flaking areas. The flaked paint layer was retouched using "Restaura Art" Restoring Colours in degrees that resembled the original [22]. After the paint layer was completely dry, the surface of the painting was re-varnished using semi-glossy Beva Finishing Varnish [23]. Modern synthetic varnishes are more stable than natural varnishes [24]. After being remounted on the new support, the paintings were retouched, re-varnished and framed, as shown in Figure 13.

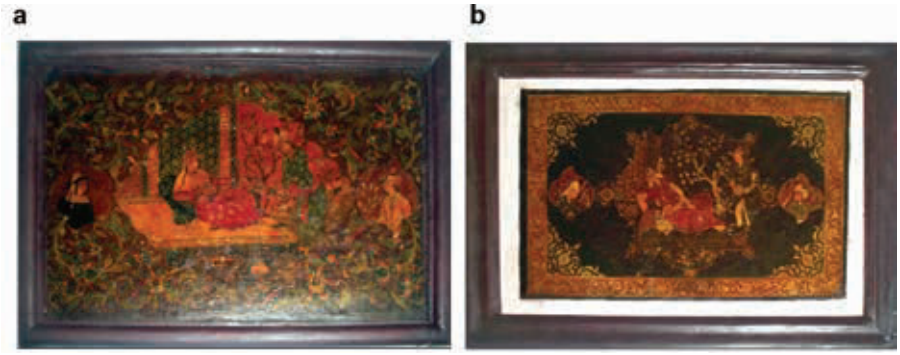

Figure 13. a) Persian Style miniature painting, no. (A) of 100/6 from the Faculty of Applied Arts Museum, Giza, Egypt, after conservation.; b) Central Asia book cover painting, no. (A) of 100/6 from the Faculty of Applied Arts Museum, Giza, Egypt, after conservation.

To protect the paperboard from the high relative humidity found in Egypt, Wacker BS 1001 was used. Wacker BS 1001 is a solvent free, silane / siloxane emulsion. This material is widely used with inorganic materials such as stones, but its effects on the properties of organic materials is unknown [25]. After testing Wacker BS 1001 on samples from the paperboard, it was observed that total impregnation forms a homoge-

nous layer on the paper fibers, thus isolating the paperboard from relative humidity and water. Changes in the mechanical properties of the paperboard were also observed. However, it was decided to impregnate a polyester sheet with Wacker BS 1001 (1:4), which was then glued to the back of the painting.

6. Conclusion

These paintings were investigated using non-destructive methods. The supports of both paintings are made of paperboard and an *alla-prima* technique is used. The artists' work is monochrome in tone. Lead red, madder, tannin and logwood are the main pigments employed in these paintings. The oil used in these paintings is safflower oil. Wacker BS 1001 is a suitable material to protect the back of these paintings from high relative humidity. Suitable materials and methods were used for the conservation and treatment of the two paintings.

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Biographical notes

Moustafa Attia Mohie, is a professor in the Department of Conservation, Faculty of Archaeology, Cairo University, Egypt. He was formerly the head of the conservation department. He is also the founder and director of the laboratory for verifying authenticity and against the forgery of historical antiquities. He has Patent no. 190808 from the European Union. He is the author of more than 20 publications in national and international scientific journals. He has also been involved in several research projects concerning the conservation field and taken part in international conferences as well as in Egypt. He has participated in the conservation of several international paintings. He has participated in the conservation of several paintings in Egypt. He has taught on several training courses for conservators at the Supreme Council of Antiquities and police officers at the Agency for verification of criminal evidence. He was the winner of one of the 5 projects selected from among 65 projects presented at Cairo University.

Mahmoud Sayed Korany, is a teaching assistant in the Conservation Department, Faculty of Archaeology, South Valley University, Luxor branch. He is a lecturer in the Department of Tourism and Archaeology, Faculty of Arts and Humanities, Jazan University, KSA. He is a conservator in the Ministry of Antiquities, Egypt. His main research interests are restoration and conservation of archaeological materials, investigation and analysis of archaeological materials, restoration and conservation of pictorial paintings, preventive conservation, condition surveys, conservation plans/ strategies, collection care surveys. He has participated in the editing of the "*Annals du service des antiquities de l'Egypte*". He has over twelve years experience as a conservator working on a range of movable and immovable artifacts. He has participated in several archaeological excavations. He is the author and co-author of 3 publications covering the conservation of cultural heritage. He is a co-investigator in 2 Scientific Research projects.

Summary

This research includes a study on the conservation of two miniature paintings produced by unknown artists and probably dated to the second half of the 18th century. These paintings are registered at the Museum of Helwan University, Faculty of Applied Arts, Giza, Egypt, No. (A) of 100/6.

This research also includes a historical and artistic study of the paintings. To study the materials and techniques used in their execution, several methods were used. XRD and XRD-EDAX were used for the examination and analysis of inorganic materials; in the case of organic materials, FTIR, UV was used to identify the pigment materials, glue, varnish, isolation layer or condition of the paper support. The oil medium in the paint layer was identified by the GLC method. Cross sections of both paintings were examined to find out about the technical knowledge used in constructing the painting / pictorial layers.

Finally, this research involves a study of how to separate two oil paintings executed on one paper support, and the most important procedures needed to carry out this step, as well as those necessary for reconstructing the painting layers. In addition, a new method is described to treat cracks.

Another new method was used to protect the paper support of these paintings from high relative humidity. This research also proposes a new tear mending method for paper objects instead of the traditional methods used in international paper and manuscript conservation laboratories.

Riassunto

È stato condotto lo studio storico-artistico e conservativo di due dipinti in miniatura realizzati da artisti sconosciuti e probabilmente risalenti alla seconda metà del XVIII secolo. Questi dipinti sono registrati presso l'Università di Helwan, Facoltà di Arti Applicate, Giza, Egitto. Allo scopo di studiare le tecniche artistiche, sono state impiegate le strumentazioni XRD, XRD-EDAX per l'analisi dei materiali inorganici; FTIR, UV per l'analisi di materiali organici ovvero, di pigmenti, colla, vernice, strato di isolamento del supporto cartaceo. Il legante ad olio nello strato di vernice è stato identificato grazie alla tecnica GLC. Sono state analizzate le sezioni trasversali di entrambi i dipinti, allo scopo di conoscere il film pittorico.

Inoltre, lo studio è stato rivolto al metodo di separazione dei due dipinti ad olio eseguiti su un supporto cartaceo e alle procedure necessarie per ricostruire gli strati di pittura, nonché ad un nuovo metodo per trattare le screpolature. Ci si è posto anche il problema, riguardante la protezione del supporto cartaceo di questi dipinti dalla umidità relativa e a quello degli strappi degli oggetti di carta, alternativo ai metodi tradizionali utilizzati per i manoscritti e documenti cartacei.